

# LC/MS Application Note

## PFAS in Cosmetics

A Comparative Analysis of Sample Preparation Techniques  
for PFAS Analysis



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## A Comparative Analysis of Sample Preparation Techniques for PFAS Analysis

### Authors

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ENVIRONMENTAL



FOOD SAFETY

### Abstract

The presence of per- and polyfluoroalkyl substances (PFAS) in cosmetics is a significant and growing concern for regulators and consumers alike. Analyzing these “forever chemicals” is notoriously difficult, as the complex waxes, oils, and pigments in products like mascara interfere with detection. This application note explores a comparative study that tackles this challenge head-on by evaluating two powerful sample preparation techniques: Solid Phase Microextraction (SPME) and Micro Solid Phase Extraction ( $\mu$ SPE) for analyzing eight key PFAS in mascara by Liquid Chromatography with Mass Spectrometry (LC/MS) [1]. The investigation revealed that the two methods offer complementary strengths. SPME proved more sensitive for water-soluble PFAS and offered a simpler, filtration-free workflow. In contrast, automated  $\mu$ SPE excelled for oil-soluble PFAS but required a sample filtration step [1]. The detection of regulated PFAS in commercial mascaras underscores the real-world applicability of these methods [1]. As the cosmetics industry navigates tightening global regulations [4, 5], this work provides a crucial guide for laboratories, demonstrating how SPME and  $\mu$ SPE serve as robust and essential tools for ensuring product safety.

### Keywords

Automation, SPME Arrow, Micro-SPE, PFAS

### Additional resources

[PAL System Content Hub](#)

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[Micro-SPE](#)

[PFAS Analysis](#)



## Introduction

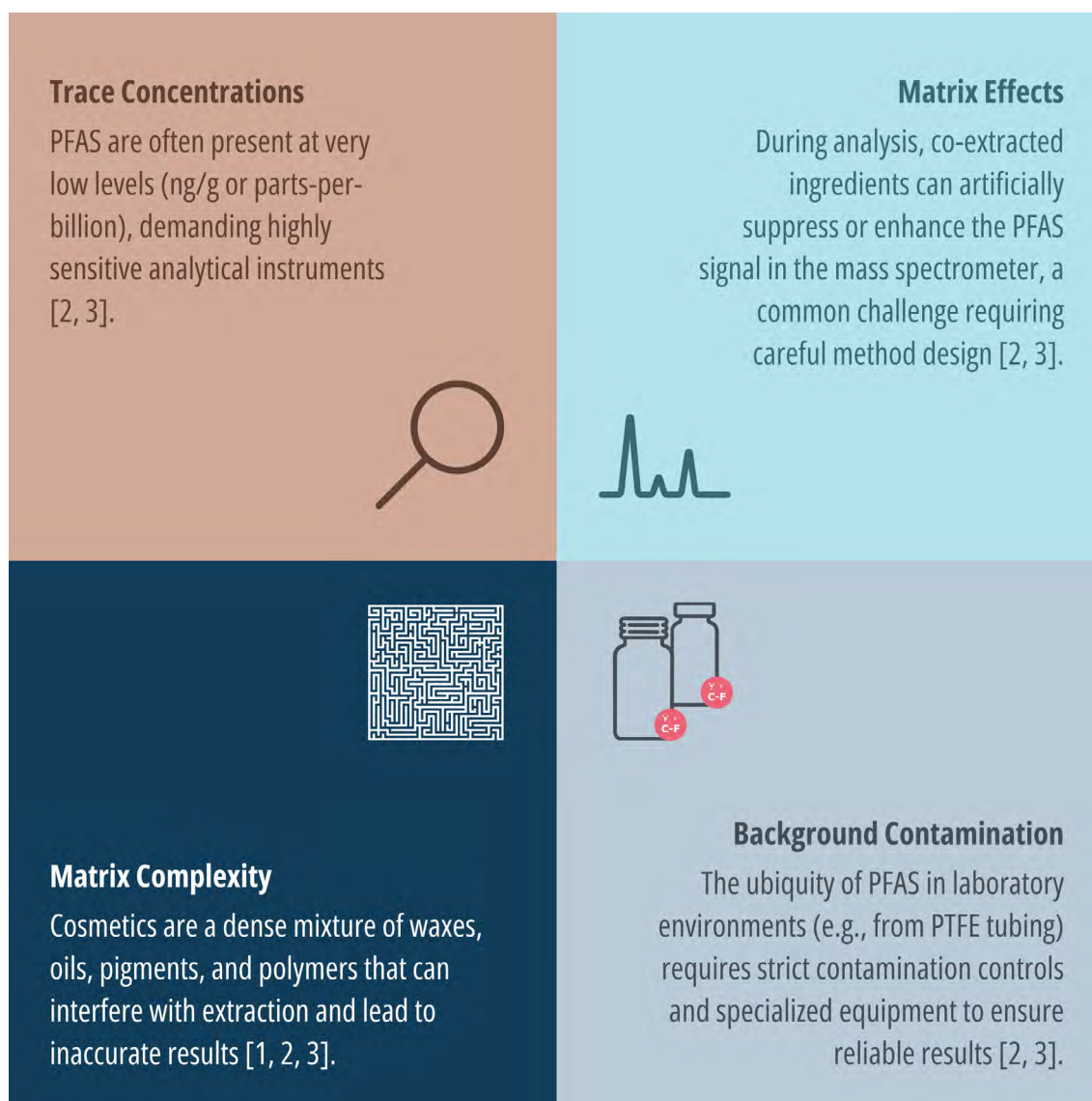
### The “Forever Chemical” Dilemma in Cosmetics

Per- and polyfluoroalkyl substances (PFAS) are a vast family of over 10,000 synthetic compounds renowned for the unparalleled strength of their carbon-fluorine (C-F) bonds [4, 6]. This chemical resilience makes them highly resistant to heat and degradation, properties that have led to their widespread use in everything from non-stick cookware to firefighting foams since the 1940s [4].

A key application for PFAS is within the cosmetics and personal care industry [7]. Certain PFAS, like Polytetrafluoroethylene (PTFE), are intentionally added to products such as foundation, lipstick, and mascara to improve water resistance, durability, and texture [4, 8, 9]. However, PFAS can also appear as unintentional contaminants from raw materials or manufacturing processes [4, 7].

The very stability that makes PFAS so useful is also the source of major environmental and health concerns, earning them the moniker “forever chemicals” [4, 5]. Their resistance to breakdown leads to their accumulation in soil, water, and wildlife, eventually making their way to humans through food, drinking water, and direct product use [2, 4, 5]. Cosmetics represent a direct exposure route, often applied to sensitive areas like the eyes and mouth [4]. A growing body of evidence links legacy PFAS, such as perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS), to a range of adverse health effects, including immune system disruption, liver damage, and increased cancer risks [2, 4, 5].

Analyzing PFAS in complex matrices like cosmetics presents several analytical hurdles (Figure 1).



**Figure 1: Key Analytical Challenges in PFAS Analysis.** The complex nature of cosmetic matrices, combined with trace analyte concentrations, potential for matrix effects, and pervasive background contamination, requires specialized and robust analytical methods.

Liquid Chromatography coupled with tandem Mass Spectrometry (LC/MS/MS) is the gold standard for PFAS analysis due to its sensitivity and selectivity [2, 3]. However, its success hinges on effective sample preparation.

While traditional methods exist, modern miniaturized techniques like Solid Phase Microextraction (SPME) and automated Micro Solid Phase Extraction ( $\mu$ SPE) offer superior sensitivity, higher throughput, and reduced solvent consumption, aligning with Green Analytical Chemistry principles [1]. This application note details the findings of a comparative study by Olomukoro et al. (2024), which evaluated these two techniques for extracting PFAS from mascara (Figure 2) [1].

## Analytical Methodologies

### A Closer Look at SPME and $\mu$ SPE

Understanding the core principles of SPME and  $\mu$ SPE is key to appreciating their distinct advantages for analyzing PFAS in challenging samples like mascara.

#### Solid Phase Microextraction (SPME)

SPME is a microextraction technique where analytes are extracted from a sample by partitioning onto a thin layer of sorbent material coated on a solid support (e.g., a fiber). As an equilibrium-based method, it elegantly integrates sampling, extraction, and concentration into a single, solvent-minimized step [3].

#### Application and Automation

In the referenced study, SPME Fibers were coated with a mixed-mode sorbent (HLB-WAX) that uses both ion-exchange

and hydrophobic interactions to capture anionic PFAS [1]. For the study, the workflow was performed manually: the SPME Fiber was immersed directly into the mascara-water dispersion for 60 minutes, after which analytes were desorbed using a small volume of solvent for LC-MS/MS analysis [1]. It is important to note that the entire SPME workflow, from extraction to desorption, can be fully automated using a PAL System. This combines the benefits of a simple, filtration-free workflow with the high precision and unattended operation of an automated system.

#### Micro Solid Phase Extraction ( $\mu$ SPE)

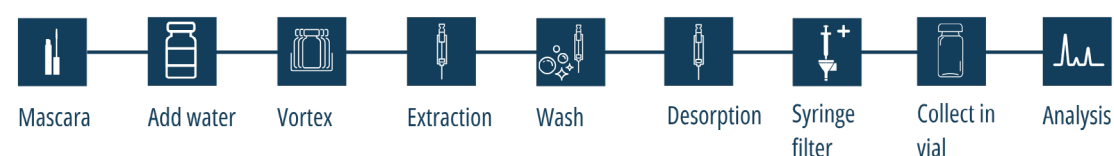
$\mu$ SPE operates on the same principles as conventional Solid Phase Extraction (SPE) but utilizes a miniaturized format, such as a small, sorbent-packed cartridge or pipette tip. The workflow involves conditioning the sorbent, loading the sample to bind the analytes, washing away interferences, and eluting the purified, concentrated analytes [1, 2].

#### Application Example

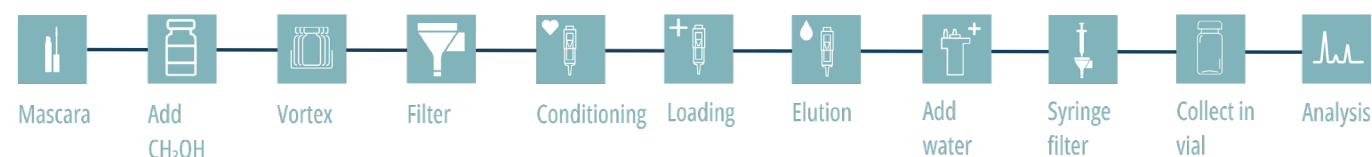
The study employed commercial weak anion-exchange (WAX)  $\mu$ SPE cartridges, which were highly effective at retaining the target PFAS [1]. The core workflow was automated on a PAL System.

A critical difference from the SPME workflow in this study was the requirement to filter the mascara dispersion (in methanol) before loading it onto the automated system. This was necessary to prevent the cartridges and autosampler components from clogging [1]. The final eluate was then analyzed by LC/MS/MS. A detailed comparison of the key features of each method is provided in Figure 3.

#### SPME Fiber Workflow



#### $\mu$ SPE Workflow



**Figure 2: Step-by-Step Workflows for SPME and  $\mu$ SPE.** A visual comparison of the manual SPME workflow (top) and the automated  $\mu$ SPE workflow (bottom) for the extraction of PFAS from mascara samples, as detailed in Olomukoro et al. (2024) [1].



## SPME (Fiber)

### Extraction

Equilibrium Partitioning

### Sample Pre-treatment

No filtration required

### Optimal Dispersion Solvent

100% Water

### Sensitivity (LOQ)

Better for hydrophilic PFAS (e.g., PFBS)

### Device Reusability

Reusable Fiber



## Micro-SPE Cartridges

### Extraction

Exhaustive Extraction (SPE)

### Sample Pre-treatment

Filtration of sample is required

### Optimal Dispersion Solvent

100% Methanol

### Sensitivity (LOQ)

Better for hydrophobic PFAS (e.g., PFOS)

### Device Reusability

Single-use Cartridges

### Key Advantage

A simplified workflow with no filtration for hydrophilic targets

### Key Advantage

High reproducibility and sensitivity for key hydrophobic targets.

**Figure 3: Method Comparison Summary.** Key differences between the SPME and  $\mu$ SPE methods as applied in the study, highlighting their complementary advantages in sensitivity, sample pre-treatment, and reusability [1].

### The Automation Note

Both workflows benefit significantly from automation on a PAL System. SPME Fibers as well as Arrows can be fully automated for unattended batch processing. The core steps of  $\mu$ SPE are automated, and the initial filtration step can also be integrated into the workflow for a complete walk-away solution.

## Key Experimental Findings

The study by Olomukoro et al. resulted in several key findings that are critical for labs analyzing PFAS in complex cosmetics. A crucial factor was the choice of dispersion solvent, which proved to be highly method-dependent. Automated  $\mu$ SPE recovery was highest when mascara was dispersed in 100% methanol, a solvent that effectively dissolves the sample matrix. In contrast, SPME extraction was most efficient in 100% water, an environment that maximizes the hydrophobic interactions driving PFAS onto the sorbent fiber. This fundamental difference highlights that the two methods rely on distinct extraction mechanisms, a vital consideration for method development. Validation of both methods showed good linearity and precision, but their sensitivity was complementary.

SPME provided lower limits of quantification (LOQ) for hydrophilic PFAS like PFBS (0.025 ng/g), while automated  $\mu$ SPE was superior for more hydrophobic compounds like PFOS (0.2 ng/g). This result demonstrates that the optimal method choice depends on the specific PFAS targets of interest. The methods were successfully applied to nine commercial mascara samples, confirming the presence of PFOA and quantifying 6:2 diPAP in four of the products. These detections underscore the real-world applicability of the methods for product safety monitoring. Finally, a major practical distinction was the need for sample filtration for the automated  $\mu$ SPE method, a manual step required to prevent clogging.

The SPME workflow, however, did not require any filtration, simplifying the overall process. Importantly, both workflows can be further automated. Steps such as liquid handling and vortexing are readily available on a PAL System.

## Conclusion

This work highlights a detailed comparative study [1] and places its findings within a broader scientific and regulatory context. It demonstrates that both SPME and automated  $\mu$ SPE are effective and complementary techniques for analyzing anionic PFAS in the highly complex mascara matrix.

- SPME offers a simple, filtration-free workflow that is ideal for analyzing hydrophilic PFAS. When automated on a platform like the PAL System, its batch processing capabilities make it a powerful, high-throughput, and reproducible option.
- Automated  $\mu$ SPE provides a sensitive and precise solution for analyzing hydrophobic PFAS, though it requires an offline filtration step for this matrix in the current workflow.

The choice between the methods depends on the specific analytical goals, including the target PFAS profile and desired level of automation. The results confirm the presence of regulated PFAS in commercial products, underscoring the importance of robust analytical methods for the cosmetics industry. As global regulations continue to evolve, sensitive and reliable techniques like SPME and  $\mu$ SPE are essential tools for ensuring product safety, demonstrating regulatory compliance, and protecting consumer health.

For a complete description of the experimental parameters, validation data, and detailed results, please refer to the original publication:

Olomukoro, A. A., Lüthy, L., Flug, T., & Gionfriddo, E. (2024). **Evaluation of Extraction Methodologies for PFAS Analysis in Mascara: a comparative study of SPME and automated  $\mu$ SPE.** *Analytical and Bioanalytical Chemistry*.

## The Evolving Regulatory Landscape

The development of robust analytical methods is timely, as regulatory pressure on PFAS in consumer products is increasing globally.

In the European Union (EU): A broad restriction covering approximately 10,000 PFAS is being pursued under the REACH chemical regulation. This would impact their use in cosmetics [4]. Specific legacy compounds like PFOA, PFOS, and PFHxS are already heavily restricted or banned under the POPs Regulation or REACH [4].

In the United States (US): The FDA is assessing PFAS safety in cosmetics under the Modernization of Cosmetics Regulation Act of 2022 (MoCRA) [10]. Concurrently, the EPA has set very low, non-enforceable health advisories for PFOA and PFOS in drinking water, signaling significant health concerns [5, 11]. While a federal ban is pending, numerous states have enacted their own laws banning intentionally added PFAS in cosmetics, with most taking effect in 2025-2026 [10].

In Asia: Nations like China are also taking decisive action. Reflecting a global trend, PFAS pollution sources in China are shifting from legacy compounds toward newer alternatives [12]. In response, PFOA and PFOS have been added to China's list of 'Strictly Restricted Toxic Chemicals,' which severely controls their use and disposal [12].

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## Equip you PAL System for PFAS Analysis

### PFAS-free Consumables



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